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<p>(21) International Application Number: PCT/SE94/00199 (22) International Filing Date: 10 March 1994 (10.03.94) (30) Priority Data: 9300955-3 23 March 1993 (23.03.93) SE (71) Applicant (for all designated States except US): BEROL NOBEL AB [SE/SE]; S-444 85 Stenungsund (SE). (72) Inventors; and (75) Inventors/Applicants (for US only): JOHANSSON, Ingegård [SE/SE]; Bogårdesgatan 8, S-416 54 Göteborg (SE). DAHLGREN, Lennart [SE/SE]; Mjölmarvägen 4, S-444 95 Ödsmål (SE). (74) Agent: ANDERSSON, Rolf; Berol Nobel AB, S-444 85 Stenungsund (SE).</p>		<p>(81) Designated States: CA, FI, JP, NO, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  Published With international search report.</p>
<p>(54) Title: ALKYL GLUCOSIDE AND USE THEREOF</p> <div style="text-align: center; margin: 20px 0;"> <math display="block">\begin{array}{c} \text{R}^1 \\ \diagdown \\ \text{CHCH}_2\text{O}(\text{G})_x\text{H} \\ \diagup \\ \text{R}^2 \end{array} \quad (I)</math> </div> <p>(57) Abstract</p> <p>In an alkyl glucoside of formula (I) R<sup>1</sup> is an alkyl group having 2-5 carbon atoms, preferably 2-4 carbon atoms; R<sup>2</sup> is an alkyl group having 4-7 carbon atoms, preferably 5 or 6 carbon atoms, the sum of the carbon atoms in R<sup>1</sup> and R<sup>2</sup> being 7-11, preferably 7-9; G is a monosaccharide residue; and x is 1-4, preferably 1 or 2. The use of the alkyl glucoside as a surfactant in a cleaning composition is also disclosed.</p>		

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## ALKYL GLUCOSIDE AND USE THEREOF

The present invention relates to a new alkyl glucoside presenting an advantageous combination of good cleaning power and low foaming, which renders it particularly suitable for cleaning hard surfaces.

In recent years, attention has focused on alkyl glucosides, since these have proved to be more easily biodegradable than other non-ionic surfactants, such as ethylene oxide adducts of fatty alcohols. US Patent Specification 3,839,318 thus describes the production of alkyl glucosides and alkyl oligosaccharides, such as n-octyl glucoside, n-hexyl glucoside, n-decyl glucoside, n-dodecyl glucoside, isodecyl glucoside, isoundecyl glucoside, isotridecyl glucoside and the corresponding oligosaccharides.

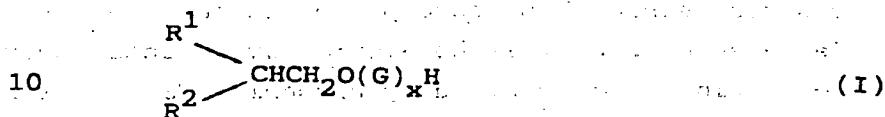
The United States Stationary Invention Registration H171 states that alkyl glucosides of formulae  $R(OG)_x$  and  $R(OG)_x$  are excellent surfactants. In these formulae, R is an alkyl or alkenyl group which is branched at the second carbon atom or at a higher carbon atom, the branch being selected from the group methyl, ethyl, isopropyl, n-propyl, butyl, pentyl, hexyl and mixtures thereof, provided that R contains from about 7 to about 30 carbon atoms; G is a saccharide group selected from the group glucose, fructose, mannose, galactose, talose, allose, altrose, idose, arabinose, xylose, lyxose, ribose and mixtures thereof; and x is 2 or more. Example 1 contains a description of the production of two product mixtures substantially made up of 2-ethylhexyl glucoside and isooctyl glucoside, respectively.

DE 20 36 472, EP 306 650, EP 306 651 and EP 366 652, inter alia, also describe alkyl glucosides.

Even though alkyl glucosides generally are easily biodegradable, they are only used to a limited extent in many ranges of application, such as the cleaning of hard surfaces, since they are too high-foaming and/ or have a poor cleaning power. Also, alkyl glucoside products con-

taining branched alkyl groups often have a disagreeable smell. It is therefore a desideratum to provide non-ionic surfactants which are about as easily biodegradable, but which have a better cleaning power and/or are more low-foaming than known alkyl glucosides.

According to the invention, it has now surprisingly been found that an alkyl glucoside of the general formula



wherein  $\text{R}^1$  is an alkyl group having 2-5 carbon atoms, preferably 2-4 carbon atoms;  $\text{R}^2$  is an alkyl group having 4-7 carbon atoms, preferably 5 or 6 carbon atoms, the sum of the carbon atoms in  $\text{R}^1$  and  $\text{R}^2$  being 7-11, preferably 7-9; G is a monosaccharide residue, and x is 1-4, preferably 1 or 2, has good cleaning and wetting properties and is low-foaming compared with other alkyl glucosides of approximately the same chain length. Compounds of formula (I) in which  $\text{R}^1$  is an alkyl group having 3 carbon atoms,  $\text{R}^2$  is an alkyl group having 5 carbon atoms, and G is a glucose residue, are especially preferred. The glucosides according to the invention do not have any disagreeable smell. In addition, they have been found to be easily degradable and have low biotoxicity. Tests have not shown any skin irritations caused by the alkyl glucosides.

The inventive compounds can be produced in conventional manner by reacting an alcohol of formula



wherein  $\text{R}^1$  and  $\text{R}^2$  are as indicated above, with a monosaccharide, the molar ratio of the alcohol to the monosaccharide being 2:1-80:1, in the presence of an acid catalyst.

The catalyst may be an inorganic or organic acid. The reaction is carried out under vacuum at 90-120°C for about 1-4 h. Conveniently, the resulting reaction mixture is first filtered and then neutralised with an organic and/or inorganic base. Finally, excess alcohol is carefully removed, e.g. by distillation, if so desired.

The alcohols of formula (II) can be obtained by a Guerbet reaction starting from n-pentanol, n-hexanol or mixtures of n-pentanol and n-hexanol, n-pentanol and n-butanol, n-hexanol and n-butanol, and n-hexanol and n-pentanol, or by an aldol condensation of the corresponding aldehydes. Preferably, the alcohol of formula (II) is 2-propylheptanol. The monosaccharide used as reactant suitably is pentose and hexose. Specific examples of monosaccharides used in the production of the inventive glucosides are glucose, mannose, galactose, talose, allose, maltose, idose, arabinose, xylose, ribose and lyxose. Glucose is usually preferred for commercial reasons.

The inventive alkyl glucosides are suitably used as surfactants in cleaning compositions, e.g. for degreasing hard surfaces or washing up. Excellent results are obtained in the degreasing of lacquered or unlacquered metal surfaces. Apart from the inventive alkyl glucoside, these compositions preferably contain a water-soluble solubiliser and suitably contain a complexing agent.

Examples of solubilisers are alkyl ether polyalkylene glycol, such as monobutyl diethylene glycol; glycols, such as diethylene glycol, dipropylene glycol and propylene glycol; alcohols, such as ethanol, propanol and isopropanol; alkyl glucosides in which the alkyl group has 4-18 carbon atoms; and/or tertiary or quaternary amine alkoxylates, in which the alkyl group, which may be straight or branched, saturated or unsaturated, has 8-20 carbon atoms, and 6-30 mol of alkylene oxide is added per mol of amine. Preferably, 50-100 mol per cent of the added alkylene oxide consists of ethylene oxide, while the remaining amount preferably consists of propylene oxide or

a mixture of propylene oxide and butylene oxide. The different alkylene oxides can be added randomly or in blocks. If the cleaning composition should be low-foaming, the alkylene oxide chain suitably ends with an addition of 1-5 mol of propylene oxide and/or butylene oxide. Usually, the ratio of solubiliser to the inventive alkyl glucoside is 1:10-5:1, preferably 1:3-3:1.

The complexing agent may be a conventional inorganic or organic complexing agent, such as an inorganic phosphate or NTA, EDTA, citric acid or a polycarboxylate. The amount added may vary from nothing at all to 300% by weight of the inventive alkyl glucoside. Preferably, the quantity ratio of the complexing agent to the alkyl glucoside is 1:10-2:1.

The cleaning compositions may further contain other additives, such as pH-adjusting agents, antifoaming agents, enzymes, other surfactants and scents. The compositions are usually aqueous and in the form of emulsions, microemulsions or solutions.

The invention will be further illustrated by the following Examples.

#### Example 1

An alkyl glucoside was produced by reacting 3 mol of 2-propylheptanol with 0.45 mol of glucose in the presence of 0.015 mol of sulphuric acid as catalyst at 110°C and 70 mbar. The reaction was interrupted after 65 min. The resulting product mixture was treated by distilling off excess alcohol under vacuum. The yield was 50 g, consisting of 74% of 2-propylheptyl monoglucoside, 15% of 2-propylheptyl diglucoside and a residue of higher oligomers. The glucosides had an average degree of polymerisation (DP) of about 1.3. The structure was determined by gas chromatography, mass spectrometry and NMR.

#### Example 2

As in Example 1, 2-butyloctanol was reacted with glucose. The reaction temperature was 112°C, and the reaction time was 90 min. The average DP was 1.5.

**Example 3**

Here, 20 ml of each of the cleaning compositions below, diluted with 10 parts by weight of water per part by weight of the composition, was applied to a vertically arranged iron sheet soiled with mineral oils, soot, salts and clay. After application, the coated surface was rinsed with water without any mechanical treatment.

Components	Composition, % by weight									
	1	2	3	4	A	B	C	D	E	
Glucoside (Example 1)	5	5	5	5						
Glucoside (Example 2)				5						
Glucoside A			8.5		5					
Glucoside B						5				
Glucoside C							5			
Glucoside D								5		
Butyldiethylene glycol		11		11	11	11	11	11	11	
Quaternary ethoxy- lated fatty amine (Berol 555)	4									
NTA	5	5	3	3	3	3	3	3	5	
Water	86	79	83.5	81	81	81	81	81	84	

Glucoside A = 2-ethylhexyl-O(G)<sub>x</sub>H

Glucoside B = isooctyl-O(G)<sub>x</sub>H

Glucoside C = n-dodecyl/n-tetradecyl glucoside (Plantaren, APG-600, Henkel)

Glucoside D = n-decyl glucoside (Lutensol GD-70, BASF)

G = glucoside residue and x = 1.5 (average value).

The resulting cleaning effect was assessed with respect to the area of the cleaned surface, as well as the actual cleanness of this surface, the figure 1 indicating no improvement and the figure 10 indicating a perfectly clean surface. The following results were obtained.

Composition	Cleaned surface, cm <sup>2</sup>	Cleanness
1	88	8
2	120	8
3	128	8
4	112	8
A	0	1
B	80	4
C	48	6
D	72	6
E	0	1

The foaming of the different ready-to-use solutions was measured according to Ross-Miles ASTM D 1173-53. The following results were obtained.

Composition	Foam height, mm	
	Instantaneously	After 5 min
1	19	3
2	23	5
3	8	5
4	30	7
A	7	0
B	20	3
C	67	63
D	46	45

These results show that the alkyl glucosides according to the invention have an excellent cleaning power and are clearly superior to alkyl glucosides having a straight carbon chain with 10-14 carbon atoms, while at the same time showing an acceptable degree of foaming. The composition containing alkyl glucosides having an alkyl group with 8 carbon atoms shows an unsatisfactory cleaning power.

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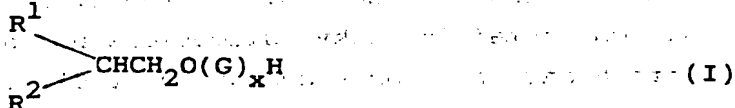
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## C L A I M S

1. An alkyl glucoside of the general formula

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- wherein  $R^1$  is an alkyl group having 2-5 carbon atoms,  $R^2$  is an alkyl group having 4-7 carbon atoms, the sum of the carbon atoms in  $R^1$  and  $R^2$  being 7-11, G is a monosaccharide residue, and x is 1-4.
2. An alkyl glucoside as set forth in claim 1, characterised in that  $R^1$  is an alkyl group having 2-4 carbon atoms, and  $R^2$  is an alkyl group having 4-6 carbon atoms, the sum of the carbon atoms in  $R^1$  and  $R^2$  being 7-9.
3. An alkyl glucoside as set forth in claim 1 or 2, characterised in that  $R^1$  is an alkyl group having 3 carbon atoms and  $R^2$  is an alkyl group having 5 carbon atoms.
4. An alkyl glucoside as set forth in claim 1, 2 or 3, characterised in that G is a glucose residue.
5. An alkyl glucoside as set forth in any one of claims 1-4, characterised in that x is 1 or 2.
6. The use of an alkyl glucoside as set forth in claims 1-5 in a cleaning composition.
7. Use as set forth in claim 6, characterised in that the cleaning composition, in addition to the alkyl glucoside, contains a water-soluble solubiliser and optionally an organic or inorganic complexing agent.
8. Use as set forth in claim 7, characterised in that the solubiliser consists of alkyl ether polyglycols, glycols, alcohols, and/or tertiary and/or quaternary alkylamine alkoxyates.

9.

Use as set forth in any one of claims 6-8, characterised in that the cleaning composition contains a solubiliser in an amount of 1:3-3:1, based on the weight of the alkyl glucoside, and a complexing agent in an amount of 1:10-2:1, based on the weight of the alkyl glucoside.

10. Use as set forth in any one of claims 6-9 of the cleaning composition for degreasing lacquered or unlacquered metal surfaces.

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## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 94/00199

## A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C07H 15/04, C11D 1/66

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C07H, C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 3839318 (RICHARD C. MANSFIELD), 1 October 1974 (01.10.74), see col. 1, 2, and 3 --	1-10
A	DE, B2, 2036472 (ATLAS CHEMICAL INDUSTRIES INC.), 4 February 1971 (04.02.71), see col. 3 and 4 --	1-10
A	EP, A2, 0387912 (KAO CORPORATION), 19 Sept 1990 (19.09.90), see part. page 1 and 2 -----	1-5

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

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**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

28/05/94

International application No.  
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Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US-A-	3839318	01/10/74	NONE		
DE-B2-	2036472	04/02/71	FR-A-	2055596	07/05/71
			GB-A-	1277516	14/06/72
			US-A-	3772269	13/11/73
EP-A2-	0387912	19/09/90	JP-A-	2247193	02/10/90
			US-A-	5130420	14/07/92

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